## UK Patent Application (19) GB (11) 2 117 666

- (21) Application No 8206863
- (22) Date of filing 9 Mar 1982
- (43) Application published 19 Oct 1983
- (51) INT CL<sup>3</sup> B01F 3/08
- (52) Domestic classification B1V C C1A G11 PD2B U1S 1609 1631 B1V
- (56) Documents cited GB 1383471
- (58) Field of search
- (71) Applicants
  University of Manchester
  Institute of Science and
  Technology
  (Great Britain),
  P.O. Box 88,
  Sackville Street,
  Manchester M80 1QD
- (72) Inventor: Graham Arthur Davies
- (74) Agent and/or
  Address for Service
  Batchellor Kirk and Eyles,
  2 Pear Tree Court,
  Farringdon Road,
  London EC1R ODS

## (54) Emulsification

(57) Emulsification is effected continuously in at least two steps. The first step involves emulsification with continuous feed of the liquid phases so as to produce an intermediate emulsion containing droplets of Interior phase dispersed throughout an exterior phase substantially all of which are of size lying within the range of from about 20 to about 30 micrometres. The intermediate emulsion is then continuously fed to a final emulsification stage in which it is subjected to further shear conditions so as to produce a final emulsion containing interior phase droplets dispersed in the exterior phase substantially all of which are less than 10 micrometres in diameter. Such a continuous emulsification process can be included in a continuous "liquid membrane" extraction process, e.g. for hydrometallurgical extraction of copper.

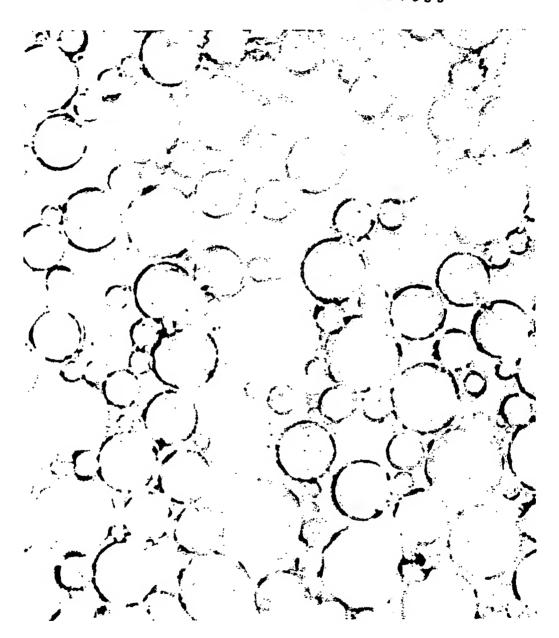


Fig. /.

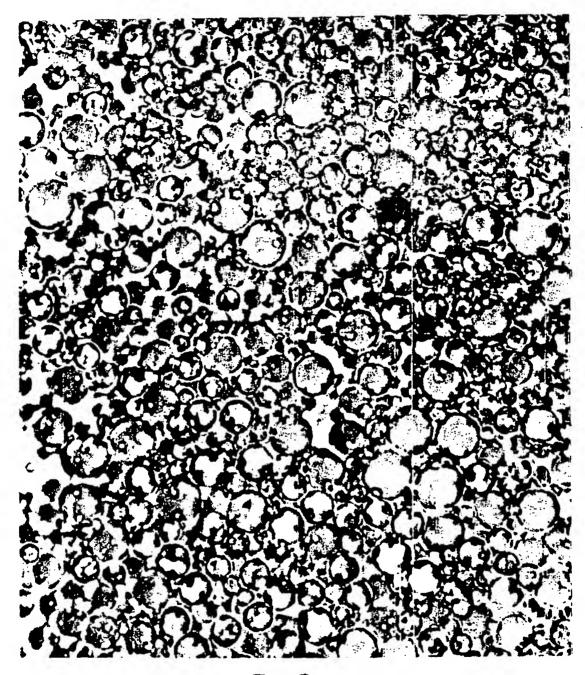


FIG. 2.

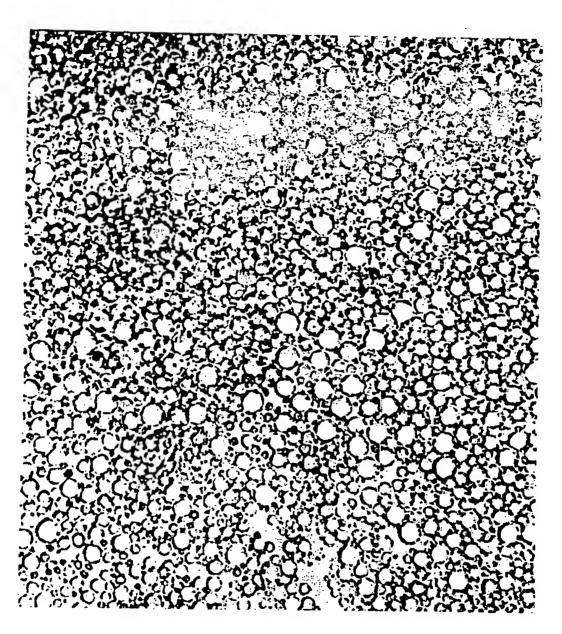


FIG.3.

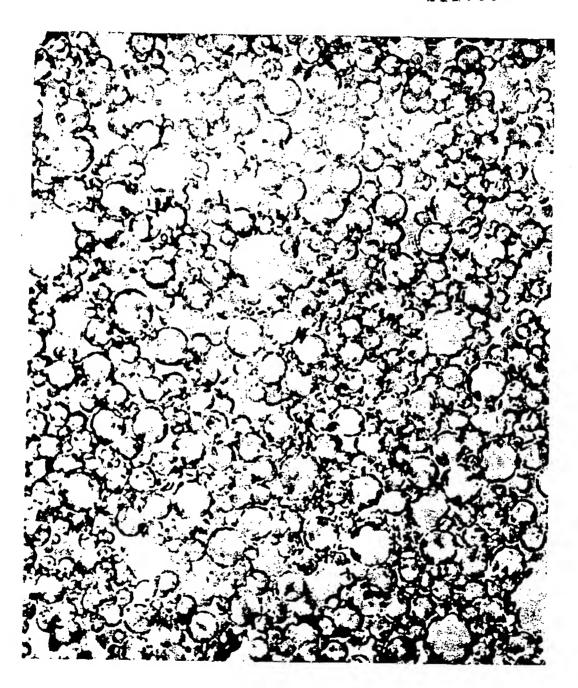


FIG. 4.

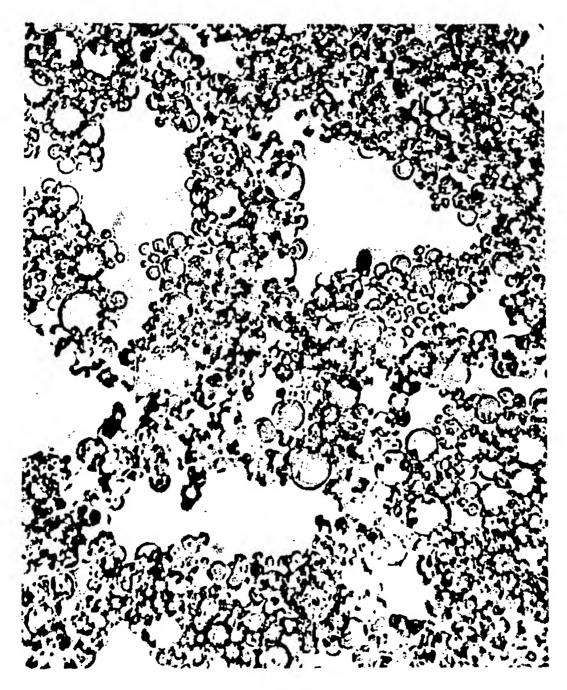
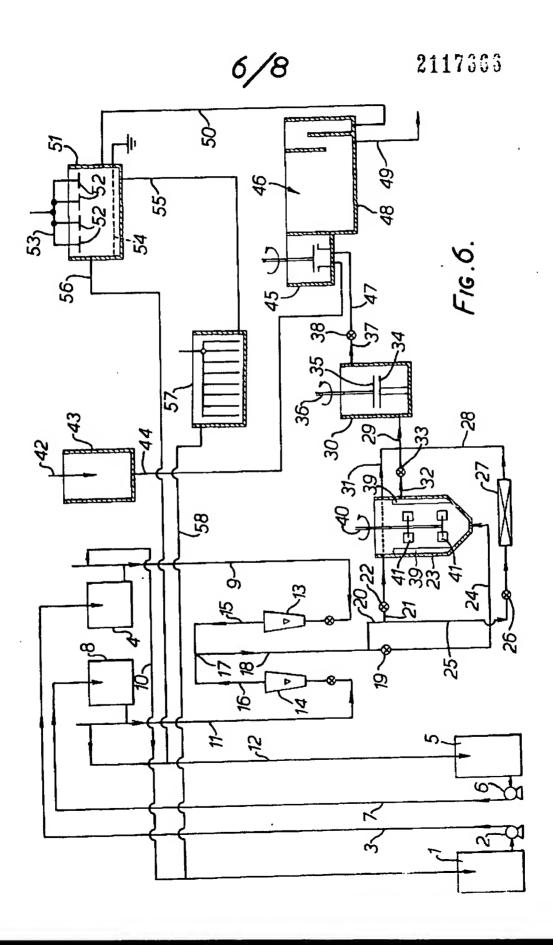


Fig.5.



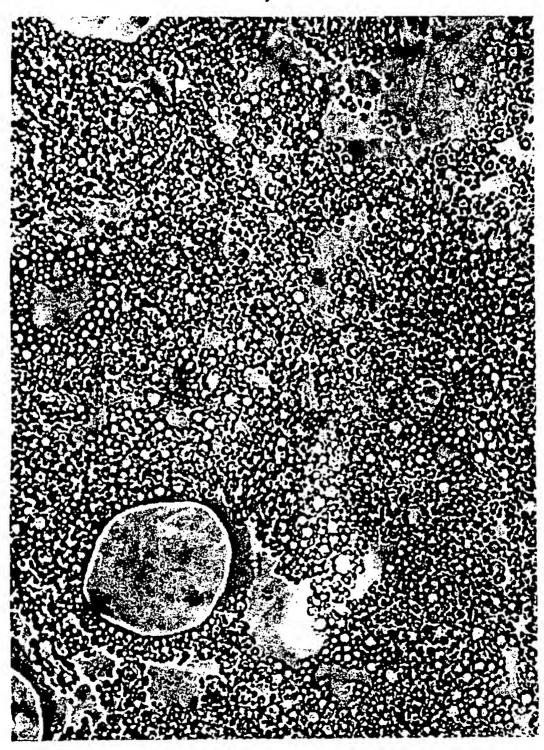


Fig. 7.

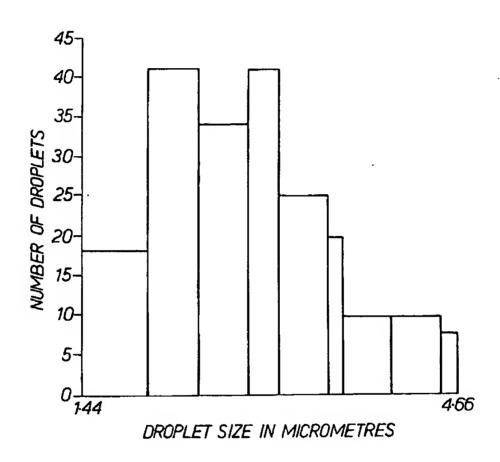


FIG.8.

## **SPECIFICATION**

## **Emulsification**

This invention relates to emulsification.

When two immisible liquids are subjected to shear conditions, as for example by being stirred with an impeller or turbine or by being forced through a so-called "static mixer", a dispersion of droplets of one of the phases (the "disperse phase") dispersed in the other phase (the "continuous phase") is formed. Such a dispersion may or may not disengage wholly or partially back into its two constituent phases upon standing under the influence of gravity.

Dispersions wherein the droplets are of a size such that substantially all of the droplets of disperse phase disengage upon standing under gravity so as

to form two layers, each consisting of a corresponding disengaged phase, may be termed "primary 20 dispersions". On the other hand, dispersions wherein the droplets do not disengage substantially completely upon standing under gravity may be termed "secondary dispersions". Generally speaking, primary dispersions contain droplets all of a diameter

25 larger than about 100 micrometres. In "secondary dispersions" the droplet size is usually about 20 micrometres or less. Under given shear conditions a dispersion containing droplets both of "primary dispersion" size and droplets of "secondary dispersion" size may be formed, of which only the primary dispersion-sized droplets disengage upon standing under gravity.

If the system includes an emulsifying agent (or a surface-active agent or wetting agent as the substances are alternatively called), an emulsion may be formed. Generally speaking, the disperse phase droplet size in an emulsion is smaller than in a dispersion and the emulsion is stabilised by the presence of the emulsifying agent. Typically, emulsion droplet sizes range from about 30 micrometres in diameter downwards, e.g. down to about 0.5 micrometres or less.

Although emulsion formation is disadvantageous in many industrial processes, emulsions are sometimes deliberately formed. For exemple, emulsion polymerisation is practised on a large scale industrially. Another process in which emulsions are deliberately formed is the so-called "liquid membrane" extraction technique. This technique was 50 pioneered by Li and his co-workers. For further teaching regarding this technique reference should be made to United States Patent Specifications Nos. 3779907, 3454489, 3389078, 3410794 3617546, 3637488, 3650091, 3696028, 3719590, 3733776, 55 3740315, 3740329, 3897308, 3942527, 3959173, 3969265, 4014785 and 4001109, as well as to United States Reissue Patent Nos. 27888 and 28002. Papers by Li and his co-workers on the subject of liquid membranes have also appeared in the literature, for 60 example in Eng. Process Des. Develop, Vol. 10, No. 2, 1971, pages 215 to 221, Separation Science, 9 (6), pages 505 to 519, 1974 and Trans. Amer. Soc of Artif.

Int. Organs, 1976, page 605 et seq.

In a typical "liquid membrane" process designed, 65 for example, for extraction of copper from a dilute solution thereof containing, for example, 250 ppm copper, measured as copper metal, a moderately concentrated sulphuric acid solution containing, for example, about 200 g.p.l. sulphuric acid (the "in-

70 terior phase"), is emulsified in a first step in an organic hydrophobic exterior phase comprising an organic solvent (e.g. kerosene) containing dissolved therein a non-ionic emulsifying agent, preferably having an HLB ratio of at least 4, and a copper

75 extraction agent (e.g. the material sold as "S.M.E. 530" by Shell International Chemicals). The resulting water-in-oil type emulsion is then contacted with the dilute copper-containing feed solution. During this contact step copper is transferred through the

80 hydrophobic phase in the form of a copper chelete and is then released into the Interior phase. At the interior phase/exterior phase interface hydrogen ions take the place in the chelete of the copper ions released into the interior phase and are transported.

35 in turn across the liquid hydrophobic phase membrane, being released into the feed solution as hydrogen ions from the chelating agent which can then form fresh chelate with copper ions from the feed solution. After this contact step the aqueous,

90 essentially copper-free raffinate is separated from the copper-laden emulsion. The raffinate is discarded and the emulsion is then broken, releasing the exterior phase and the copper-containing interior phase. The interior phase contains perhaps

about 150 g.p.l. copper in the form of copper sulphate and may then be passed to an electrolysis stage. Spent acid from the electrolysis stage can then be re-emulsified in the exterior phase and the resulting emulsion re-cycled to the contact stage.

The above-described copper extraction process is but one example of a liquid membrane process.
 Moreover, it is not essential that the interior phase of the emulsion should be an aqueous phase. An example of a liquid membrane process in which
 oil-in-water type emulsions are used, with an organic interior phase and an aqueous exterior phase, is described inter alia in the afore-mentioned United
 States Reissue Patent Specification No. 27888.

For successful operation of a liquid membrane
extraction process, it is essential that the emulsion
should be sufficiently stable during the contact step
for "bleeding" of interior phase into the feed solution to be minimal and yet should be readily broken
in order to release the loaded interior phase for
recovery of the extracted species. Hence in the case
of the copper extraction process described above it
is essential that the water-in-oil type emulsion
should be sufficiently stable during contact with the
dilute aqueous copper-containing feed solution for
"bleeding" of acid to be minimal whilst being
capable of being readily broken, after separation of
the copper-loaded emulsion from the raffinate, in
order to release the copper-leden acidic aqueous

interior phase for electrolysis or other copper recov-

ery steps. These two requirements at first sight are in conflict one with another. As Li recognises in United States Patent Specifications Nos. 4001109 (see column 2, line 58 to column 3, line 23) and 4125461 (see column 2, line 44 to column 3, line 18), the formulation of suitable emulsions is difficult and their breaking is problematic. Li goes on to state that a variety of known processes, including electrostatic precipitation, are completely unsuitable for breaking 10 such emulsions.

Although Li teaches that emulsion breaking cannot be accomplished by electrostatic coalescence, it has been shown that, by careful control of the emulsion forming conditions so that an emulsion is 15 formed in which the droplets of interior phase have a uniformly small droplet size, it is possible to minimise "bleeding" of interior phase from the emulsion during the contact step, whilst enabling ready breaking of the emulsion by electrostatic coalescence 20 after the contact step has taken place. For further details of this electrostetic coalescence procedure, reference should be made to British Patent Specification No. 2000754A and United States Patent Specification No. 4283290, as well as to the paper "The 25 Extraction of Copper from Dilute Aqueous Solutions using a liquid membrane process", by T. P. Martin and G. A. Davies, Hydrometallurgy, 2 (1976/77), pages 315 to 334.

Various types of homogeniser are commercially
30 available, some being recommended for laboratory
use whilst others are designed for industrial application. Such homogenisers are recommended in some
cases for batch preparation and in others for operation with a continuous mixed liquid phase feed.

35 In the course of experimentation leading up to the making of the present invention, it was found that commercially available homogenisers recommended by their manufacturers for continuous emulsification do not in practice produce emulsions suitable, for example, for use in the liquid membrane technique and with the desirable droplet size distribution recommended in British Patent Specification No. 2000754A and United States Patent Specification No. 4283290. In all cases it proved impossi-

45 ble with the equipment tested, as supplied, to produce emulsions in a continuous manner with satisfactory properties for use with the liquid membrane technique of the afore-mentioned British Patent Specification No. 2000754A and United States
 50 Patent Specification No. 4283290 even though batch scale tests using a laboratory homogeniser had

resulted in the production from the same organic

and aqueous solutions of emulsions possessing the required droplet size distribution and the required 55 characteristics. Upon microscopic examination of these continuously prepared emulsions it was found that, compared with the batch emulsion experiments, there was present in each of the continuously formed emulsions a small but significant proportion

60 of large diameter droplets of interior phase. Even when the residence time of the phases in the continuous hornogeniser was increased there was still formed in each case a similarly high undesirable proportion of these larger size droplets of interior 65 phase. It would accordingly be desirable to provide a method of continuously forming an emulsion having a relatively uniform small droplet size, for example the desirable droplet size distribution taught by the 70 afore-mentioned British Patent Specification No. 2000754A and United States Patent Specification No. 4283290.

The present invention accordingly seeks to provide an improved method for continuous emulsifica75 tion whereby emulsions may be reliably formed in controlled manner containing a relatively uniform small droplet size distribution of the disperse phase droplets.

According to the present invention there is pro-80 vided a method of continuous emulsification which comprises continuously dispersing one in the other in one or more initial emulsification stages in the presence of an emulsifying agent an aqueous phase and an organic hydrophobic phase substantially 85 insoluble in the equeous phase under shear conditions such as to form an intermediate emulsion containing droplets of interior phase dispersed throughout an exterior phase substantially all of which are of a size lying within the range of from 90 about 20 to about 30 micrometres, continuously passing resulting intermediate emulsion to a final emulsification stage, subjecting intermediate emulsion in the final emulsification stage to shear conditions such as to form a final emulsion contain-95 ing droplets of interior phase dispersed in the exterior phase substantially all of which are less than 10 micrometres in diameter, and continuously recovering resulting final emulsion from the final emulsification stage.

100 The invention also relates to a continuous extraction process for continuously recovering a dissolved species from a feed solution thereof which comprises contacting e feed stream of the feed solution with a final emulsion comprising droplets of an interior 105 phase substantially all of which are less than 10 micrometres in diameter dispersed in an exterior phase which is substantially insoluble in the feed solution and also in the interior phase, contains an emulsifying agent dispersed therein, and is perm-110 able to the desired species, separating a raffinate from the resulting loaded emulsion whose interior phase is now loaded with the desired species, breaking the loaded emulsion, recovering the desired species from the interior phase released upon 115 breaking the emulsion, emulsifying interior phase now depleted in the desired species in the exterior phase in one or more initial emulsification stages to form an intermediate emulsion containing droplets of interior phase dispersed throughout the exterior 120 phase substantially all of which are of a size lying within the range of from about 20 to about 30 micrometres, continuously passing resulting intermediate emulsion to a final emulsification stage, and subjecting intermediate emulsion in the final 125 emulsification stage to shear conditions such as to form the final emulsion.

Preferably the droplets of interior phase in the final emulsion are substantially all of a size lying in the range of from about 0.3 to about 10 micrometres with the majority of the droplets being distributed

over the range of from about 0.8 to about 3 micrometres. The or each initial emulsification stage may comprise a static mixer (e.g. a Sulzer, Kenics or Ross static mixer), a continuously stirred tank (e.g. 8 Davy "Pump Mix" reactor), a pump mixer, or a turbine mixer (e.g. a Plenty or Silverson mixer). Although more than one initial emulsification stage may be used, each operating under progressively more severe shear conditions so as progressively to 10 reduce the Interior phase droplet size from one stage to the next, it will normally suffice to use a single initial emulsification stage, provided that this is of appropriate design. Provision may be made for recirculating emulsion from the exit end of the or each 15 initial emulsification stage, or from the exit end of one or more of the initial emulsification stages, if there is more than one such stage, to the inlet end of that stage or of another such stage. For example, intermediate emulsion can be recirculated from the 20 exit end of the last one of a plurality of initial emulsification stages to the inlet end of the first one of those stages.

The final emulsification stage preferably comprises a homogeniser adapted for continuous supply of liquids and for continuous removal of emulsion. The final emulsification stage should normally be, and preferably is, operated without recirculation of emulsion.

The aqueous and organic hydrophobic phases are supplied to the initial emulsification stage, when there is only one initial emulsification stage, in the proportion desired in the final emulsion, e.g. in the ratio of from about 5:1 to about 1:5 by volume, e.g. about 1:1 by volume. When more than one initial emulsification stage is used, the phases can be delivered to the first one of the initial emulsification stages in the proportion desired in the final emulsion or in a different proportion therefrom, in which latter case further quantities of one of the phases may be supplied as necessary to a subsequent initial emulsification stage, or to several such stages, as appropriate, in order to produce in the intermediate emulsion the desired phase ratio.

The organic phase may comprise, for example, a 45 polymerisable monomer, whilst the aqueous phase comprises the ingradient normally present therein an emulsion polymerisation system. Alternatively the aqueous and organic phases may be formulated so as to be suitable for use in a liquid membrane 50 process. For further teaching as to suitable components and their proportions to be used in liquid membrane techniques reference should be made to the aforementioned United States Patent Specifications and papers in the name of Li and his co-wor-55 kers, as well as to British Patent Specification No. 2000754A to United States Patent Specification No. 4283290 and to the paper by T. P. Martin and G. A. Davies, the disclosure of all of which documents is to be regarded as incorporated herein by reference. In 60 forming the intermediate emulsion the choice of shear conditions during dispersion of the phases once in the other is crucial to formation of an emulsion in which substantially all of the interior

phase droplets are of a size within the range of 20 to

65 30 micrometres. When the intermediate emulsion is

formed using one or more static mixers, the shear conditions will depend upon the design of the static mixer or mixers and the flow rate of the liquid phases therethrough. When the intermediate emulsion is formed using a stirred tank reactor or other form of agitated mixer (or a plurality of such reactors and mixers), then the shear conditions will be determined largely by the dwell time in the emulsification zone, as well as on the speed of rotation of the agitator.

Similar considerations apply to the shear conditions to be used in the final emulsification stage which is typically a continuous feed homogeniser. In this case the shear conditions can be controlled by selecting an appropriate feed rate of intermediate emulsion to the final emulsification stage, by varying the rate of rotation of the rotatable homogenizer disc or discs, or by eltering the spacing between the discs, or by a combination of two or more of these expedients.

The selection of appropriate shear conditions can readily be accomplished by a process of trial and error whilst monitoring the droplet size distribution in the intermediate emulsion or in the final emulsion, as the case may be.

Breaking of the loaded emulsion in the continuous extraction process of the invention can be accomplished by electrostatic coalescence as taught more fully in British Patent Specification No. 2000754A and United States Patent Specification No. 4283290.

The invention will be further described with reference to the accompanying drawings, wherein:

Figures 1, 2 and 3 are photomicrographs, each at a magnification of 1450x, of a water-in-oil type emultion sion formed in a batch emulsifier after shear times of 4,7 and 12 minutes respectively;

Figures 4 and 5 are photomicrographs, each at magnification of 1450x, of emulsions prepared in commercially available continuous feed single stage 105 homogenisers;

Figure 6 is a block diagram of an apparatus utilising the method of the invention;

Figure 7 is a photomicrograph of an emulsion, at a magnification of 1450x, made using the apparatus of 110 Figure 6 at a feed rate of 900 ml/min; and

Figure 8 is a histogram showing the droplet size distribution of the emulsion of Figure 7.

Referring to the drawings, Figures 1 to 3 show that in a batch emulsification process, the interior phase 115 droplet size decreases with increasing residence time in the homogeniser. As can be seen from Figure 3 the resulting emulsion has a relatively narrow distribution of interior phase droplet sizes. Such an emulsion can be used successfully in the liquid 120 membrane process described in British Patent Specification No. 2000754A and United States Patent Specification No. 4283290 as well as in the paper by T. P. Martin and G. A. Davies. The apparatus used to make the emulsions of Figures 1 to 3 was a 125 Janke-Kunkel Ultra-Turrax batch homogeniser. In each case the aqueous phase was a 100 g/I H<sub>2</sub>SO<sub>4</sub> solution whilst the organic phase contained 10% w/v S.M.E.530 copper extractant (sold by Shell Interna-

tional Chemicals) and 1% w/v Span 20 oil-soluble 130 surfactant dissolved in Escaid 100 (sold by Esso Chemicals UK Ltd.) as solvent. The phase ratio was 1:1 by volume. The emulsion of Figure 3 was found to give acceptable results in the liquid membrane process of British Patent Specification No. 2000754A and United States Patent Specification No. 4283290.

The emulsions of Figures 4 and 5 were obtained from the same feed solutions as were used in making the emulsions of Figures 1 to 3, also at a phase ratio of 1:1 by volume. The emulsion of Figure 10 4 was of the water-in-oil type and was obtained using a homogenizer, Silveson model, at a flow rate of 500 ml/min emulsion. Although the majority of the droplets are small and relatively uniform in size, there are a small, but significant number of interior 15 phase droplets of diameter exceeding about 10 micrometres. This emulsion is unsatisfactory for use in the liquid membrane process of the aforementioned British Patent Specification No. 2000754A and

20 Figure 5 is a similar microphotograph of an emulsion, prepared in a Janke-Kunkel, Ultra-Turrax flow chamber model, continuous homogeniser also using a flow rate of 500 ml/min. This emulsion was also found unsatisfactory for the afore mentioned

United States Patent Specification No. 4283290.

25 liquid membrane process. Again it contained a small, but significant, number of interior phase droplets exceeding about 10 micrometres in diameter. (The large irregular patches visible in Figure 5 are air bubbles). Figure 6 illustrates a preferred form 30 of apparatus with which the method of the invention may be used, as well as a modification thereof.

Aqueous phase (i.e. 100 g/l H<sub>2</sub>SO<sub>4</sub> solution) is pumped from storage tank 1 by way of pump 2 and line 3 to an aqueous header tank 4. Organic phase 35 (i.e. 10% w/v S.M.E.530 copper extractant plus 1%

(i.e. 10% w/v S.M.E.530 copper extractant plus 1% w/v Span 20 in Escaid 100) is pumped from storage tank 5 by pump 6 through line 7 to organic header tank 8. From aqueous header tank 4 aqueous phase is drawn off through line 9 at a desired rate, whilst

40 excess aqueous phase is recycled to feed tank 1 through overflow line 10. In a similar way organic phase is withdrawn in line 11 from organic header tank 8, whilst the remainder is recycled to feed tank 5 by way of overflow line 12. After passing through 45 respective flow meters 13, 14 the aqueous and

respective flow meters 13, 14 the aqueous and organic phases pass on in lines 15, 16 to junction 17. The combined stream flows on in line 18. By

control of valve 19 in the flow of mixed liquid phases can be directed either via lines 20 and 21 and then
50 through valve 22 to the upper end of a pre-mixer unit 23 or through line 24 to the lower end of premixer unit 23. By closing valves 19 and 22 the mixed liquid phases can be passed through line 25 and valve 26 to an in-line blender 27 of the static mixer type.

55 Emulsion can be recovered from in-line blender 27 by means of line 28 and passed on in line 29 to a continuous homogeniser 30. Alternatively emulsion can be withdrawn from premixer unit 23 at an upper level via line 31 or at a slightly lower level through

60 line 32, controlled by valve 33 and passed on via line 29 to homogeniser 30. Reference numeral 31 indicates the static disc of the homogeniser whilst reference numeral 35 indicates the rotating disc of the homogeniser which is mounted on shaft 36.

65 Emulsion can be recovered from homogeniser 30 in

line 37, the flow rate through homogeniser 30 being controlled by means of valve 38.

Premixer unit 23 is fitted with a number of vertical stationary baffles 39, only two of which are visible in 70 Figure 6, and the contents are stirred by means of a shaft 40 fitted with two turbine blade impeller 41 each fitted with six turbine blades. Shaft 40 is driven by means of a variable speed electric motor (not shown).

75 Shaft 36 of homogeniser unit 30 may also be driven by a variable speed motor which may be fitted with a thyristor control. It will be appreciated that homogeniser unit 30 is shown diagrammatically and that the vertical spacing between discs 34 and 80 35 has been greatly exaggerated for the sake of clarity.

The aqueous solution to be treated, for example a copper ore leach liquor, is fed via line 42 to holding tank 43 and thence via line 44 to a mixing compartment 45 of a conventional mixer-settler 46. Emulsion from homogeniser unit 30 is also fed to mixing compartment 45 via line 47. The flow rates of aqueous feed solution through line 44 and of emulsion through line 47 are adjusted to give the 90 desired residence time in the mixing compartment 45 of mixer-settler 46 and the desired emulsion:feed solution ratio, e.g. about 1:1.

From mixing compartment 45 the resulting dispersion consisting of globules of emulsion dispersed in a continuous aqueous phase is fed to settling compartment 48 of mixer-settler 46. Bulk separation of the phases occurs in settling compartment 48 and the aqueous phase is discharged via line 49. This raffinate can be recycled to the leaching site after addition of the appropriate amount of make-up acid.

agglomerate and are discharged from settling compartment 48 via line 50, which leads to an electrostatic coalescer 51. This contains a number of electrodes 52 which are connected to a bus-bar 53 which is maintained at a high positive potential. Reference numeral 54 illustrates an earthed ground electrode. In the electrostatic coalescer the droplets of interior phase coalesce and the coalesced interior phase is withdrawn via line 55 whilst the exterior phase is withdrawn via line 56 and returned via line 12 to holding tank 5 for recycling.

The globules of emulsion in the dispersion

The coalesced interior phase in line 55 then passes to a conventional electrolysis cell 57 where electrowinning is carried out in a conventional manner.

Spent liquor discharged from cell 57 is recycled to holding tank 1 via line 58.

Figure 7 is a photomicrograph at a magnification of 1310x of an emulsion prepared using the apparatus of Figure 6 as obtained in line 37. Figure 8 is a histogram of interior phase droplet sizes showing the interior phase droplet size distribution of the emulsion of Figure 7. These results are summarised in the following Table:

T-	ъ	1	•
	-	-	•

W-mbaw		classes		9	
-					
Class	(mi	crometres)	-	Prequency	
1.44	-	1.99		18	
1.99	-	2.41		41	
2.41	-	2.84		34	
2.84	-	3.11.		41	
3.11	-	8.54		25	
3.54	_	3.67		20	
3.67	-	4.09		10	
4.09	-	4.59		10	
4.52	-	4.66		8	
Mean		2.9	9260 micrometre	98	
Standa	rđ				
Deviat	ion	.74	160		
Skewne	88	.3	549		
Rurtos	is	48	881		
Number of droplets					
counte	đ		207.		

As can be seen from Figure 8 and the Table the droplets substantially all lie in the range from about 0.5 to about 10 micrometres, whilst the majority of the droplets lie in the range of from about 0.8 micrometres to about 3 micrometres. This emulsion is eminently suitable for use in the liquid membrane process of British Patent Specification No. 2000754A and United States Patent Specification No. 4283290. CLAIMS (Filed on 28/Feb/83)

- 10 1. A method of continuous emulsification which comprises continuously dispersing one in the other in one or more initial emulsification stages in the presence of an emulsifying agent an aqueous phase and an organic hydrophobic phase substantially
- 15 insoluble in the aqueous phase under shear conditions such as to form an intermediate emulsion containing droplets of interior phase dispersed throughout an exterior phase substantially all of which are of a size lying within the range of from
- 20 about 20 to about 30 micrometres, continuously passing resulting intermediate emulsion to a final emulsification stage, subjecting intermediate emulsion in the final emulsification stage to shear conditions such as to form a final emulsion contain-
- 25 ing droplets of interior phase dispersed in the exterior phase substantially all of which are less than 10 micrometres in diameter, and continuously recovering resulting final emulsion from the final emulsification stage.
- A method according to claim 1, in which the droplets of interior phase in the final emulsion are substantially all of a size lying in the range of from about 0.3 to about 10 micrometres with the majority of the droplets being distributed over the range of from about 0.8 to about 3 micrometres.
  - A method according to claim 1 or 2, in which the or each initial emulsification stage comprises a static mixer, a continuously stirred tank, a pump mixer or a turbine mixer.
- 4. A method according to any one of claims 1 to 3, in which a single initial emulsification stage is used.
- A method according to any one of claims 1 to
   in which emulsion is recirculated from the exit end
   of the or each initial emulsification stage, or from the

exit end of one or more of the initial emulsification stages, if there is more than one such stage, to the inlet end of that stage or of another such stage.

- A method according to any one of claims 1 to
   5, in which the final emulsification stage comprises a homogeniser adapted for continuous supply of liquids and for continuous removal of emulsion.
- A method according to any one of claims 1 to 6, in which the final emulsification stage is operated
   without recirculation of emulsion.
- 8. A method according to any one of claims 1 to
  7, in which the ageous and organic hydrophobic phases are supplied to the first emulsification stage or, if there are more than one initial emulsification
  50 stages, to the first of the initial emulsification stages
  - in the ratio of from about 5:1 to about 1:5 by volume.

    9. A method according to claim 8, in which the ratio is about 1:1 by volume.
- A method of continuous emulsification con-65 ducted substantially as herein described and/or as illustrated with references to the drawings.
- 11. A continuous extraction process for continuously recovering a dissolved species from a feed solution thereof which comprises contacting a feed
  70 stream of the feed solution with a final emulsion comprising droplets of an interior phase substantially all of which are less than 10 micrometres in diameter dispersed in an exterior phase which is
- substentially insoluble in the feed solution and also
  75 in the interior phase, contains an emulsifying agent
  dispersed therein, and is permeable to the desired
  species, separating a raffinate from the resulting
  loaded emulsion whose interior phase is now loaded
  with the desired species, breaking the loaded emulsion recovering the desired species from the interior
- phase released upon breaking the emulsion, emulsifying interior phase now depleted in the desired species in the exterior phase in one or more initial emulsification stages to form an intermediate emulsion containing droplets of interior phase dispersed throughout the exterior phase substantially all of which are of a size lying within the range of from about 20 to about 30 micrometres, continuously passing resulting intermediate emulsion to a final emulsification stage, and subjecting intermediate emulsion in the final emulsification stage to shear
- conditions such as to form the final emulsion.

  12. A process according to claim 11, in which the droplets of interior phase in the final emulsion are substantially all of a size lying in the range of from about 0.3 to about 10 micrometres with the majority of the droplets being distributed over the range of from about 0.8 to about 3 micrometres.
- 13. A process according to claim 11 or claim 12, 100 in which the or each initial emulsification stage comprises a static mixer, a continuously stirred tank, a pump mixer, or a turbine mixer.
- A process according to any one of claims 11 to 13, in which a single initial emulsification stage is 105 used.
- 15. A process according to any one of claims 11 to 14, in which emulsion is recirculated from the exit end of the or each initial emulsification stage, or from the exit end of one or more of the initial 110 emulsification stages, if there is more than one such

stage, to the inlet end of that stage or of another such stage.

- 16. A process according to any one of claims 11 to 15, in which the final emulsification stage com-5 prises a homogeniser adapted for continuous supply of liquids and for continuous removal of emulsion.
  - 17. A process according to any one of claims 11 to 16, in which the final emulsification stage is operated without recirculation of emulsion.
- 18. A process according to any one of claims 1 to 17, in which the aqueous and organic hydrophobic phases are supplied to the first emulsification stage or, if there are more than one initial emulsification stages, to the first of the initial emulsification
- 15 stages in the ratio of from about 5:1 to about 1:5 by volume.
  - 19. A process according to claim 18, in which the ratio is about 1:1 by volume.
- 20. A continuous extraction process conducted 20 substantially as herein described and/or as illustrated with reference to the drawings.
- 21. Each and every novel feature, and each and every novel combination of features, substantially as herein described and/or illustrated in the drawings 25 and their equivalents.

Printed for Her Majesty's Stationary Office by The Tweeddale Press Ltd., Berwick-upon Tweed, 1983.
Published at the Patent Office, 25 Southammton Buildings, London, WC2A 1AY,

from which copies may be obtained.